**Synopsis over**

**PhD thesis**

# Fabrication and characterization of nanomaterials for electromagnetic applications

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# Introduction

As technology advances day by day, the use of electromagnetic waves also increases. So far, without the use of electromagnetic waves, many parts of technology are powerless. Just as important is the use of electromagnetic waves, the protection of these waves is also important. Nowadays, nanotechnology has reached almost every stage of technology and has made enormous changes, including in electromagnetic applications. In this thesis the fabrication and characterization of nanomaterials in respect with the electromagnetic applications will be done.

# What is an electromagnetic (EM) wave?

Whenever a magnetic field interacts with an electric field, Electromagnetic (EM) waves are created. The electric field oscillates perpendicular to the magnetic field and the resulted EM wave propagation is also perpendicular to both them. EM waves have a velocity of constant $3.0 \times 10^8$ m/s while traveling in the vacuum. Electromagnetic waves travel across anything, (i.e. gas, liquid or solid material) or even vacuum dissimilar to sound waves which are mechanical waves that need an exact medium for travelling. We are surrounded with this form of energy as electromagnetic radiation which propagates through space–time and carries electromagnetic radiant energy. Activities in the human level such using as microwave to heat up a food, using GPS devices for precise location navigation or using X-rays detection in medical area are impossible without the use of electromagnetic radiation. Figure 1 shows the spectrum of electromagnetic waves with accord to their frequencies/wavelengths. The range of the electromagnetic spectrum goes from radio waves and microwaves which carry lower energy and thus longer wavelength to X-rays and gamma rays which have higher energy waves and so shorter wavelength.

# What is Electromagnetic interference?

Any kind of external radiation or conduction disturbance generated that can affect electrical circuits is known as Electromagnetic interference (EMI). The emission conducted is referred to the noise generated internally due to the weakness of

design of electrical circuits such as power cables and electrical wires and it has the range of several KHz to 30 MHz; On the other hand the radiated emission is generated externally such as the predetermined electromagnetic waves from the radio broadcasting station antenna and the inadvertent electromagnetic waves coming from transceivers with high speed.

- EMI can be caused by
    - Currents can radiate electric fields
    - Natural fields, switching circuits, etc.
    - Coupling of electric fields into circuits and components
- Effects of EMI
    - It can have temporary disturbances which can be solved
    - It can cause the loss and disruption of data
    - In long term in can bring system failure
    - In some cases it also can affect human life

EMI shielding is possible by preventing electromagnetic waves from entering an electrical system. This is done either by reflecting or by absorbing the power of the incident beam. Previously, metals were used for many cases. Aluminum and galvanized steel are the most used among the metals. Copper, zinc, nickel and silver are also used for some other purposes. As today's electronics grow smaller, lighter and faster, metals are not helpful considering the weight. In addition, electromagnetic pollution does not really disappear and does not diminish because the EM signals almost completely reflect on the surface of the metal from the environment just outside the shield. Therefore, a lot of research efforts are focused on developing EMI shielding materials with accurate absorption and reflection based on new materials that are light, corrosion-resistant, flexible, easy to process, etc.

Examples of EMI

- Cellular device interference
    - Airplanes
    - Medical equipments
- Power converters
    - Capacitive, inductive, and resistive loads
    - Caused by sudden load changes
    - Available at house (Computers, TVs, mixers, kitchen appliances etc.)
- Electromagnetic pulse
    - Intense surge of electromagnetic radiation
    - Gamma rays which trap high-energy electrons existed in earth’s magnetic field

- Radio frequency interference
    - Radio stations, two way transmitters

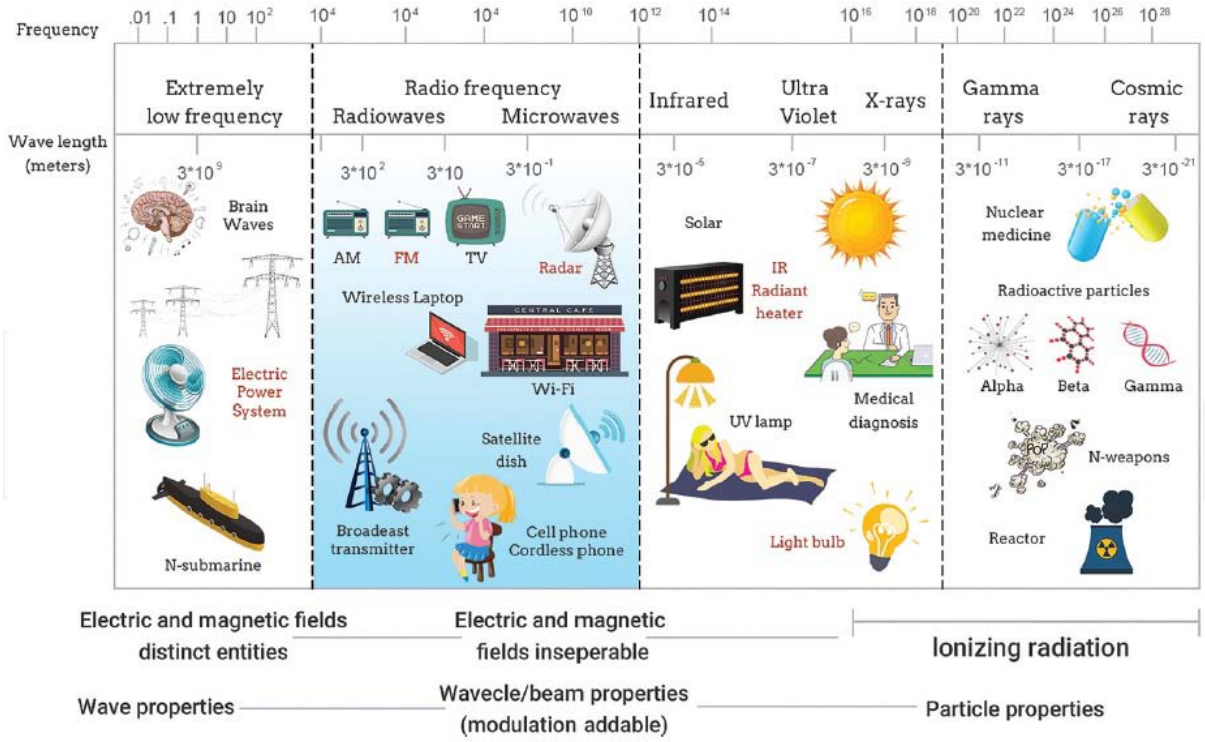


Figure (1). The EM spectrum with respect to the range of frequencies and wavelengths[17]

# Nanotechnology and EMI shielding

Over the past decades science has a huge development in the field of nanomaterials, and investigating their synthesis, fabrication, analyzing their properties, and finally finding proper applications. Nanomaterials are of great interest in research because of their inherent properties, due to their scale, which is not observed on a macro-scale.

Large surface areas are obtained from nanomaterials due to their small size, resulting in many interesting material properties different from the bulk. Atoms which are residing at the surface of the material are not basically saturated, and there are many sites whit nanomaterials that are very active chemically and unstable both in thermal and structural states. This allows higher chemical activities with melting points a lot lower. Also it allows electronic and structural defects that can induce unusual electronic and optical properties.

In addition, the size of the nanomaterials is very small and that stimulates the charges and movement within the material, causing the electron structures to be continuously discrete and to gradually change the optical spectra.
These phenomena are referred to as quantum confinement effects. This phenomena occurs whenever the nanomaterials' size matches the Bohr radius level for semiconductor nanoparticles.
Microwave absorption has turned in to an innovative field of application when nanomaterials with their inherent magnetic and dielectric properties are used. The basic properties of these materials and their interactions with gigahertz range electromagnetic irradiance are studied continuously. A lot of military and civilian systems need the pairing of different materials that can work with microwave irradiance in particular areas, such as prevention of leakage of information from communications and computing systems, and in the stealth technology the materials covering. Therefore, the scientific community must make a wide material range available for use in military and civilian goals so the needs for these particular application areas converge with the proficiency of the materials selected.
So far most of the materials which are developed for the microwave absorption purposes have the morphology of the nano-scale. Reports from various papers indicate a tendency for the magnetic and dielectric interactions of the materials as a function of the size of the particle to increase the electromagnetic interaction by decreasing the size of a particle. Many of the studies over the essential effects of magnetic and dielectric nanomaterials in absorption of microwave do not clearly inspect materials functioning based on particle size however the parameter of particle size appears to form an important role while using these nanomaterials for microwave absorption and electromagnetic interference shielding.

# Microwave absorption mechanisms

Since an electromagnetic wave is the pairing of an oscillating magnetic field and an electric field, so the materials that cause the microwave absorption, interact with one or both of these fields, thus brings the interaction between light-matter in the electromagnetic gigahertz region spectra.

Dielectric loss is the electric property that shows the cooperation happening between the nanomaterial and the electric part of an electromagnetic irradiance and its outcome is the reflection loss (RL); on the other hand, magnetic loss is the magnetic characteristic that shows the interaction between the nanomaterial and the magnetic part of the electromagnetic irradiance. Using the Maxwell's equations and the fundamental relations resulting from those equations, it is observed that a

nanomaterial response to an electromagnetic wave can be derived the permeability and permittivity of the bulk material, where permittivity (equation (1)) and permeability (equation (2)) are complex parameters that enfold the energy storage and dissipation.

$$\varepsilon_r = \varepsilon/\varepsilon_0 = \left(\varepsilon^{'} - i.\varepsilon^{"}\right) \quad (1)$$

$$\mu_r = \mu/\mu_0 = \left(\mu^{'} - i.\mu^{"}\right) \quad (2)$$

# Fundamentals of analysis

The EMI shielding can be described as a three part process, reflection, absorption and multiple reflections inside the material therefore methods of analysis will be subcategorized into two parts. First, there are methods which only concentrate on reflecting the electromagnetic radiation. Second, there are methods which investigate the electromagnetic wave transmission and reflection as well. Both methods have particular techniques that produce frames of data that adequately describe the responses givens by the material, so they can extract permeability and permeability values. Therefore from these values the reflection loss parameters can be derived.

For the first method (reflection methods), just the single scattering parameter is needed to be quantified by experiment and that can be achieved by sending and receiving a signal via the incident port (port 1) Figure (2). The scattering response obtained from the electromagnetic signal that is going back to the source of radiation through the signal port is measured as $S_{11}$ parameter. In the reflection methods we require a set of $S_{11}$ values to resolve the complex permeability and permittivity as a function of scattering parameters. For the second method (transmission/reflection), a two-port device is required to quantify the electromagnetic response for both the forward and also the backward (reflected) directions. Therefore the scattering parameters system is expanded to a $2 \times 2$ matrix.

The $S_{11}$ parameter in the $2 \times 2$ scattering matrix, still represents the response of the incident irradiance from port 1, also the subsequent emitted signals through internal reflections raised by the matching impedance of the material due to the free space. The $S_{12}$ parameter is now defined as both the response signal of electromagnetic radiation from port 1 travelling via the material and propagating towards the detector (port 2) and also any signals created from internal reflections subsequently. $S_{21}$ and $S_{22}$ parameters can be generated subsequently by examining in both directions. Various sets of algorithms are available for converting S-parameters to the wanted permeability and permittivity values. Further material properties can be described using these permittivity and permeability values.

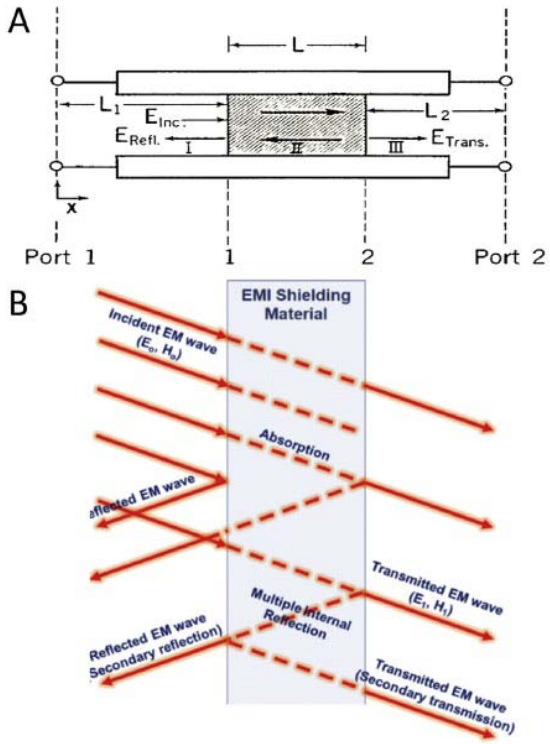


Figure (2). A) The coaxial transmission/reflection line for quantifying scattering parameters. B) Electromagnetic interaction with a given material.[18]

A tangential loss is defined here as the ratio of the loss action as for the lossless. Similar terms are also available for magnetic and electric interactions, therefore parameters of dielectric and magnetic loss tangents, shown in Equation (3). Where, $\varepsilon'$ is the complex permittivity for the lossless action and $\varepsilon''$ is the complex permittivity for the loss action. Furthermore $\mu'$ is the complex permeability for the lossless action and $\mu''$ is the complex permeability for the loss action. Finally, $\delta$ represents the angle of loss between these two components, and $tan\,\delta\varepsilon$ is the dielectric loss tangent and $tan\,\delta\mu$ is the magnetic loss tangent. The dissipating energy given a system is represented by these loss tangents. Different electric interactions via conduction and polarization that can cause electric loss will be represented by the dielectric loss tangent. Materials with a small loss tangents (tan $\delta\varepsilon \ll 1$) have a considerably small conductivity compared to the angular frequency and permittivity are considered as good dielectrics. On the contrary, materials with large loss tangents (tan $\delta\varepsilon >> 1$) are considered as good conductors. The tangent of magnetic loss is more limited due to the absence of magnetic monopoles. The peak of loss tangents usually occurs at higher frequencies compare to the peaks of relaxation. Since the lossless interaction works as a function of frequency and in the whole transfer process, the dissipation efficiency and also the rate of energy

transfer from lossless interaction to loss both depend on the value of the lossless interaction.
The quality factor, which is another term for describing material response, is obtained by inversing the parameters of loss tangents, shown in Equation (4). Where $Q\varepsilon$ is the quality factor for dielectric response and $Q\mu$ is the quality factor for magnetic response. Therefore by inversing the values of the magnetic quality factor and the dielectric quality factor and adding them together the total quality factor Q is obtained, shown in Equation (5). In general, the total quality factor Q has an inverse relation with the energy dissipation.

$$\tan\delta_\varepsilon = \frac{\varepsilon''}{\varepsilon'} \; ; \; \tan\delta_\mu = \frac{\mu''}{\mu'} \qquad (3)$$

$$Q_\varepsilon = \frac{1}{\tan\delta_\varepsilon} = \frac{\varepsilon'}{\varepsilon''} \; ; \; Q_\mu = \frac{1}{\tan\delta_\mu} = \frac{\mu'}{\mu''} \qquad (4)$$

$$\frac{1}{Q} = \frac{1}{Q_\varepsilon} + \frac{1}{Q_\mu} \qquad (5)$$

# Nanomaterials for microwave absorption

- Carbons (carbon nanotubes, carbon fibers, Graphene, Amorphous and other carbon materials)
- Carbide
- Oxides (Iron oxide, Manganese oxide, Titanium oxide, Zinc oxide, Silicon oxide, Aluminum oxide)
- Sulfides (Copper sulfide, Cobalt sulfide)
- Phosphides
- Polymers
- Metals/alloys
- Carbon nitrides
- Metal-organic frameworks

### *Carbon Nanotubes (CNTs)*

- Qj et. al. synthesized pure CNTs with CVD technique adding water soluble potassium chromate as catalyst
- The proficiency bandwidth behaves as a function of thickness

- Using doping agents such as Fe, FeCo, FeCoNi increases the magnetic interaction with the electromagnetic radiation
- Similarly multiwall carbon nanotubes (MWCNTs) are developed with dopants so that they induce microwave absorption, through the production of nanomaterials

Table 1. Microwave Absorption Performance of Carbon Nanotubes based Nanomaterials[18]

| Nanomaterials | Performance | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | d (mm) | $\Sigma\Delta f_{-10}$(GHz) | Max RL (dB) | Freq (GHz) | $\varepsilon'$at Max | $\varepsilon^{"}$ at Max | tan $\delta_{\varepsilon}$at Max | $\mu'$at Max | $\mu^{"}$at Max | tan $\delta_{\mu}$ at Max |
| Raw CNTs | 3.0 | 0 | -9.9 | 10.6 | 6.4 | 1.6 | 0.25 | 0.98 | -0.01 | -0.01 |
| Purified CNTs | 3.0 | 2.9 | -14.9 | 10.3 | 6.5 | 2.3 | 0.35 | 0.99 | -0.03 | -0.03 |
| Fe/CNT's | 1.2 | 16.0 | -18.2 | 11.8 | 28.6 | 43.1 | 1.51 | 2.02 | 1.6 | 0.79 |
| Fe/CNT's | 1.2 | 16.0 | -24.8 | 10.9 | 29.7 | 40.7 | 1.37 | 1.94 | 1.91 | 0.98 |
| FeCo/CNT's | 2.0 | 8.0 | -15.5 | 9.5 | n/a | n/a | n/a | n/a | n/a | n/a |
| FeCoNi/CNT's | 2.0 | 5.6 | -28.2 | 15.2 | n/a | n/a | n/a | n/a | n/a | n/a |
| CNT's | 3.5 | 2.9 | -22.9 | 11.4 | 33.2 | 34.9 | 1.05 | 1.0 | 0.01 | 0.01 |
| Fe/CNT's | 3.5 | 2.9 | -31.8 | 13.2 | 20.2 | 25.3 | 1.25 | 1.1 | 0.1 | 0.09 |
| Fe/CNT's | 3.5 | 4.2 | -22.7 | 15.6 | 34.0 | 55.0 | 1.62 | 0.71 | 0.03 | 0.04 |

## *Carbon Fibers (CFs)*

- Made by Chu et. al. using coaxial electrospinning
- Shows the Reflection Loss (RL) of solid CFs is greater than hollow ones
- The frequency of maximum absorption of solid materials shows asymptotically increase with respect to the outer diameter of the CFs.
- The solid fiber's proficiency bandwidth was consistent in the range of fiber diameters which had been reported with proficiency, between 2.0 and 2.3 GHz.
- As the fiber diameter was decreased in hollow CFs from 210$\mu m$, the proficiency bandwidth also decreased from 1.8 GHz to 1.1 GHz.

Table 2. Microwave Absorption Performance of Carbon Fiber based Nanomaterials[18]

| Nanomaterials | Performance | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | d (mm) | $\Sigma\Delta f_{-10}$(GHz) | Max RL (dB) | Freq (GHz) | $\varepsilon'$at Max | $\varepsilon^{"}$ at Max | tan $\delta_{\varepsilon}$at Max | $\mu'$at Max | $\mu^{"}$at Max | tan $\delta_{\mu}$ at Max |
| H1.1 μm CFs | 3.0 | 1.1 | -14.8 | 5.0 | 27.3 | 8.7 | 0.32 | 1 | 0 | 0.00 |
| H210μm CFs | 3.0 | 1.8 | -17.0 | 6.4 | 15.2 | 6.5 | 0.43 | 1 | 0 | 0.00 |
| D1.43 μm CFs | 3.0 | 2.3 | -22.9 | 7.6 | 11.7 | 4.5 | 0.38 | 1 | 0 | 0.00 |
| D7.40 μm CFs | 3.0 | 2.0 | -20.1 | 9.0 | 10.0 | 4.0 | 0.40 | 1 | 0 | 0.00 |

## *Graphene*

- Wang et al. investigated Graphene-based nanomaterials
- The graphene nanomaterial on itself was not valuable for microwave absorption as its essential interaction in gigahertz range of electromagnetic radiation showed lacking.

- However it was suggested that via doping the interaction characteristics could change the interaction of light/matter so the microwave region absorption would be induced.

### *Iron Oxide*

- Ni et al. synthesized $Fe_3O_4$

- The microspheres and the nanocrystals both illustrated high permittivity values, like the classic Magnetite because of the existence of the $Fe^{2+}$ ions inside the material.
- The values of permeability of the materials were enhanced more than the trivial values, showing to contribute for the electromagnetic absorption via magnetic interaction.

### *Zinc Oxide*

- The basic interaction between ZnO and gigahertz-range radiation lacks, since the material doesn't show any magnetic interaction or any strong dielectric action to run reflection loss.
- However, using dopants to induce electromagnetic interaction has been successful for interaction with microwaves of particular frequencies.
- Adding magnetic particles like nickel and iron induced disturbance in the response of the nanomaterial to the magnetic field of incident electromagnetic waves.

### *Silicon Oxide & Aluminum Oxide*

- Show the same behavior as Graphene and Zinc Oxide.
- $SiO_2$ compare to ZnO shows even lower reflection loss.
- The ZnO|$SiO_2$ composites show enhanced reflection loss values.
- Ceramics as the complex form of silicates as are also introduced for microwave absorption.

- Pairing aluminum oxide and magnetic materials together increases the responses to the both electric and magnetic field of the irradiance electromagnetic waves.

# Objectives

Following are the objectives for the proposed work:

nanocrystals and microspheres to study the magnetite's magnetic interactions in gigahertz range with electromagnetic radiation.

1. To explore different nanomaterials for electromagnetic applications.
2. To fabricate different sizes of electromagnetic nanomaterials.
3. To characterize the properties of nanomaterials with respect to electromagnetic applications.
4. To compare and suggest suitable solution for electromagnetic application using nanomaterials.

# Research methodology

1. To study literature to identify nanomaterials for electromagnetic application and choosing the desirable properties for the selection of the nanomaterials.
2. Using synthesis techniques such as sol-gel, precipitation, CVD, etc. to fabricate different shapes and sizes of nanomaterials from those selected above.
3. Study the properties of the fabricated materials using XRD for investigating the crystalline nature of the nano-particles. Using SEM to study the morphology of the nano-particles.
4. Comparing the results obtained from different synthesis and suggesting the application with respect to the electromagnetic properties.

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